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(54) Title: METHOD OF MAKING A FINISHED PRODUCT FROM A FEEDSTOCK, AN ALKALINE EARTH METAL OXIDE OR HYDROXIDE, AND A THERMOSETTING RESIN

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(57) Abstract: A method of making a finished product includes the steps of (a) providing a dry feedstock selected from the group consisting of particles or fibres of a lignocellulosic material; particles of an expanded mineral; particles of a foamed set hydraulic binder; particles of an undensified silica fume or a mixture of two or more thereof; (b) mixing the feedstock with: (i) a suitable amount of a thermosetting resin in finely divided dry powder form; (ii) a suitable amount of a first binding agent selected from the group consisting of an alkaline earth metal oxide or hydroxide; or a mixture of two or more thereof, in finely divided dry powder form; and (iii) a second binding agent such as a pozzolan in finely divided dry powder form; to give a starting material; (c) subjecting the starting material of step (b) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form a cohesive product; (d) providing to the cohesive product of step (c) water in an amount sufficient to permit the reaction of the first and second binding agents to form a binder; and (e) allowing the binder to set to form the finished product. The finished product may, for example, be a board.

**METHOD OF MAKING A FINISHED PRODUCT FROM A FEEDSTOCK, AN  
ALKALINE EARTH METAL OXIDE OR HYDROXIDE, AND A  
THERMOSETTING RESIN**

**BACKGROUND OF THE INVENTION**

THIS invention relates to a method of making a finished product such as a board, from a feedstock which is a suitable particulate or fibrous material, a binder, and a thermosetting resin.

Various types of particle board are known, manufactured from particles of a suitable feedstock, e.g lignocellulosic particles or fibres, bound together with either a hydraulic binder or a thermosetting resin or a combination thereof.

Lime (calcium oxide) has potential as a binder in building products provided its cure speed can be accelerated and its properties improved.

Research has been done by the Wood Research Institute of Kyoto University, Japan and the Nichiha Company Limited of Japan on accelerating the curing of

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Portland cement bound particle board, as well as improving the mechanical properties of the particle board, by post treating the pre-pressed and initially set particle board with carbon dioxide, either in super critical or gaseous form. One disadvantage of this process is that before such a treatment may be carried out, either the particle board must be pre-set which implies that the chemical reactions in the binder have already commenced, or alternatively the particle board must in some way be pressed and clamped during treatment.

### SUMMARY OF THE INVENTION

According to a first aspect of the invention there is provided a method of making a finished product including the steps of:

- (a) providing a dry feedstock selected from the group consisting of particles or fibres of a lignocellulosic material; particles of an expanded mineral; particles of a foamed set hydraulic binder; particles of an undensified silica fume; or a mixture of two or more thereof;
- (b) mixing the feedstock with:
  - (i) a suitable amount of a thermosetting resin in finely divided dry powder form;
  - (ii) a suitable amount of a first binding agent selected from the group consisting of an alkaline earth metal oxide or hydroxide or a mixture of two or more thereof, in finely divided dry powder form;
  - (iii) a suitable amount of a second binding agent selected from the group consisting of:
    - (1) a pozzolan selected from the group consisting of densified silica fume; ground granulated blast furnace slag; fly ash; and a mixture of two or more thereof;
    - (2) an unexpanded clay;
    - (3) a high silicon containing lignocellulosic material;
    - (4) silica;

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- (5) an alkali metal silicate;
- (6) aluminium oxide or hydroxide;
- and a mixture of the two or more thereof, in finely divided dry powder form;
- to give a starting material;
- (c) subjecting the starting material of step (b) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form a cohesive product;
- (d) providing to the cohesive product of step (c) water in an amount sufficient to permit the reaction of the first binding agent with the second binding agent to give a binder; and
- (e) allowing the binder to set to form the finished product.

In step (d), simultaneously or sequentially with the provision of water, but before the binder has set, there may also be introduced into the cohesive product of step (c) carbon dioxide to assist in the setting of the binder.

According to a second aspect of the invention there is provided a finished product made by the method described above.

#### **DESCRIPTION OF EMBODIMENTS**

The crux of the invention is a method of making a finished product from a feedstock, a binder formed from first and second binding agents, and a thermosetting resin.

The feedstock may be particles or fibres of a lignocellulosic material.

A lignocellulosic material refers to any plant material emanating from the photosynthetic phenomenon.

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The particles or fibres of the lignocellulosic material are preferably finely divided. For example, the fibres may be provided as unifibres or bundles of a small number of unifibres. In other words, the lignocellulosic material is broken down into single fibres or bundles of a small number of fibres. Examples of suitable finely divided lignocellulosic fibres are paper mill sludge or recycled paper waste, or the fibres used in the production of medium density or high density fibre board. Alternatively, the fibres may be sourced from agriculture such as sisal, kenaf, hemp or the like, with a length of from 5 to 12 mm inclusive, preferably from 2 to 6 mm inclusive.

When the lignocellulosic material is provided in particle form, the particles preferably have a size of from 0.1 mm to 2 mm diameter inclusive.

An important factor in the choice of the lignocellulosic material is that it should not swell on water wetting, i.e during impregnation of the cohesive product with the water.

The feedstock may also be particles of an expanded mineral such as exfoliated vermiculite, expanded perlite, an expanded clay, an alumina, a pumice or diatomaceous earth.

The vermiculite particles preferably have a particle size of 2 mm diameter or smaller, down to a particle size of 100 micron diameter.

The expanded perlite particles preferably have a particle size of 2 mm diameter or smaller, down to a particle size of 100 micron diameter.

The other expanded minerals which may be used must also have a particle size of 2 mm diameter or smaller, down to a particle size of 100 micron diameter.

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The feedstock may also be particles of a foamed set hydraulic binder, e.g a foamed hydraulic cement aggregate which is then milled or ground into particles having a size of 2 mm diameter or smaller, down to a particle size of 100 micron diameter.

The feedstock may also be particles of an undensified silica fume, having a particle size of from 5000 m<sup>2</sup>/kg to 20000 m<sup>2</sup>/kg.

The feedstock may also be a mixture of two or more of the various feedstocks mentioned above.

The preferred feedstocks are lignocellulosic particles or fibres, exfoliated vermiculite particles, expanded perlite particles, particles of undensified silica fume, or a mixture of two or all three thereof.

The feedstock is mixed firstly with a suitable amount of a thermosetting resin in finely divided dry powder form.

The thermosetting resin is preferably a novolac phenol formaldehyde resin, i.e a resin in which the molar ratio of phenol to formaldehyde exceeds parity, which is used with a suitable catalyst.

An example of a suitable catalyst for use with a novolac phenol formaldehyde resin is hexamethylene tetramine.

An example of a suitable novolac phenol formaldehyde resin catalyst combination is a two stage resin with a hexamethylene tetramine content of between 6 and 14%, with a hot plate gel time at 150°C of between 40 and 120 seconds, with a flow in mm at 125°C of between 30 and 75 mm, and with a particle size sieve analysis percentage retained on a 200 mesh screen of a maximum of 2%. Examples are the PRP Resins of South Africa, code Varcum

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7608 which may be used as a modifier for a slow curing phenolic system such as Varcum 3337, or which may be used on its own.

As indicated, the thermosetting resin must be provided in finely divided dry powder form. By finely divided there is meant that the thermosetting resin must have a particle size of 98% passing a 200 mesh screen.

The feedstock is also mixed with a suitable amount of a first binding agent.

The first binding agent is an alkaline earth metal oxide or hydroxide such as calcium oxide or magnesium oxide or hydroxide, or a mixture thereof. Preferred materials are lime (CaO) and magnesia (MgO).

The first binding agent must also be provided in finely divided dry powder form. By finely divided there is meant that the first binding agent must have a particle size of from 300 to 1000 m<sup>2</sup>/kg inclusive.

The feedstock is also mixed with a suitable amount of a second binding agent.

The second binding agent is selected from the following.

1 - A pozzolan selected from:

- densified silica fume with a particle size in the range of from 5000 to 20000 m<sup>2</sup>/kg;
- ground granulated blast furnace slag with a particle size in the range of from 300 to 2000 m<sup>2</sup>/kg;
- fly ash with a particle size in the range of from 300 to 2000 m<sup>2</sup>/kg;
- a mixture of two or more thereof.

2 - An unexpanded clay in the form of finely divided particles of hydrous silicates of aluminium, sometimes with magnesium or iron substituting for

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all or part of the aluminium. Examples are kaolinite, halloysite, illite and montmorillonite.

- 3 - A high silicon containing lignocellulosic material, i.e a lignocellulosic material containing an above average silicon content such as particles of hurds of hemp. These are the wood-like cores of the stem of the hemp plant Cannabis Sativa. Another example is defibrated rice straw.
- 4 - Silica (silicon dioxide) in finely divided form, also called silica flour.
- 5 - An alkali metal silicate such as sodium or potassium silicate. An example is SP33 by Sand Cl with a weight ratio  $S_1O_2:Na_2O$  of 3.3 to 1.0.
- 6 - Aluminium oxide or hydroxide.

The second binding agent may also be a mixture of two or more of the above.

Where not otherwise specified, the particle size of the second binding agent must be such that 98% of the particles pass a 300 mesh screen, preferably a 200 mesh screen, more preferably a 150 mesh screen.

The first and second binding agents react, in the presence of water, with each other and optionally also other components present to form a binder.

The various components of the starting material may be used in the following mass percentages to give 100% of the starting material:

- |                      |                                      |
|----------------------|--------------------------------------|
| Feedstock            | - 10% to 50%, preferably 20% to 40%, |
| Thermosetting resin  | - 1% to 20%, preferably 2% to 5%,    |
| First binding agent  | - 10% to 65%, preferably 30% to 50%, |
| Second binding agent | - 10% to 65%, preferably 30% to 50%. |

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As the product of step (c) is cohesive, i.e is held or stuck together, there is no need to press or clamp the cohesive product to hold it together during step (d).

There may also be added into the mixture of the feedstock, the thermosetting resin and the first and second binding agents additional components including inorganic filler materials in particle or fibre form, such as for example wollastonite, 200 mesh or finer, and synthetic fibres, or mixtures thereof.

It is to be noted that all the components of the starting material are used and mixed in dry form to ensure that the binder does not set before step (e).

After step (b) in which the feedstock is mixed with the thermosetting resin and the first and second binding agents, the starting material so produced is subjected to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form a cohesive product.

For example, the starting material may be pressed at a temperature of up to 200°C preferably in a range of from 120° to 200°C and a pressure of from 20 to 70kg/m<sup>2</sup> inclusive, preferably 20 to 30kg/m<sup>2</sup> inclusive, for a time of up to 15 seconds/mm thickness, or usually about 8 seconds/mm thickness, to form the cohesive product.

In this step, the thermosetting resin sets to form the cohesive product, while the first and second binding agents remain unreacted.

In step (d) of the method of the invention, there is provided to the cohesive product of step (c), water in an amount sufficient to permit the reaction of the first and second binding agents to give a binder.

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Water permits dissolution of the first binding agent, i.e calcium oxide or magnesium oxide or hydroxide, so that it can react with the second binding agent, to form, for example, calcium and/or magnesium silicate hydrates, or calcium or magnesium aluminates.

Optionally in step (d), simultaneously or sequentially with the provision of water, but before the binder has set, there may also be introduced into the cohesive product of step (c) carbon dioxide to assist in the setting of the binder.

This may be achieved in various ways.

Firstly there may be added to the cohesive product of step (c) a carbonation reagent selected from the group consisting of carbon dioxide; and an aqueous solution of a compound or compounds which release carbon dioxide; under conditions that cause release of carbon dioxide in the cohesive product.

When the carbonation reagent is carbon dioxide at super critical, near super critical or high carbon dioxide density conditions, then when this carbonation reagent is added to the cohesive product of step (c), any calcium hydroxide in the binder reacts with the carbon dioxide to form a carbonate plus free water. This water then can be used for the complete hydration of the binder. Thus in these circumstances, it may not be necessary to add additional water.

Alternatively water may be added as such, or may form a solvent or diluent for the carbonation reagent.

In this case the carbonation reagent may be carbon dioxide, e.g gaseous carbon dioxide, liquid carbon dioxide, super critical or near super critical carbon dioxide, or a solution of carbon dioxide in a solvent.

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The carbonation reagent may also be a compound which releases carbon dioxide, in solution.

The carbonation reagent may also be a combination in solution of compounds which together, under suitable conditions, release carbon dioxide.

For example, the carbonation reagent may be a carbonate or bicarbonate, in solution, such as for example carbonic acid or sodium bicarbonate.

The carbonation agent may be for example a potassium carbonate of formula  $K_2CO_3 \cdot 2H_2O$  or sodium carbonate or sal soda of formula  $Na_2CO_3 \cdot 10H_2O$ .

Step (d) is carried out under conditions that cause release of carbon dioxide from the carbonation reagent in the cohesive product. When the carbonation reagent is for example a compound which releases carbon dioxide at a particular temperature, then the cohesive product must be raised to this temperature or higher in step (d). For example, sal soda releases carbon dioxide at a temperature of about 35°C, and thus, when using sal soda as the carbonation reagent, the cohesive product should be raised to a temperature of 35°C or higher in step (d).

Other conditions which cause the release of carbon dioxide may simply be the presence of water, as with an acid/base couple, or pressure, or a combination of temperature and pressure or the like.

When using carbon dioxide itself, then it is simply required that the carbon dioxide penetrate the cohesive product.

Secondly there may be added to the starting material in step (b) a carbonation reagent which is a dry compound or dry compounds which release carbon

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dioxide under specified conditions. This compound or these compounds thus form part of the cohesive product of step (c).

In step (d) the cohesive product of step (c) is then subjected to the specified conditions to cause the release of carbon dioxide in the cohesive product.

For example the compound may be sodium carbonate or potassium carbonate, in which case the addition of water in step (d), optionally together with the application of heat, leads to release of carbon dioxide.

Alternatively the compounds may be an acid/base couple, in which case the addition of water in step (d) leads to release of carbon dioxide.

The introduction of carbon dioxide assists in the setting of the binder. It may accelerate the setting of the binder and/or may control the microstructure of the binder in the finished product.

For example the carbon dioxide propagates the rapid formation of hydration products such as calcium silicate hydrate and calcium carbonate, in a time span of from between 10 and 60 minutes, at ambient temperatures.

The use of carbon dioxide is particularly efficacious where the feedstock contains components which may interfere with the curing of the binder. For example, where the feedstock contains a lignocellulosic material, the water soluble extracts of the lignocellulosic material may inhibit the setting of the binder. The inclusion of carbon dioxide serves to lessen or overcome this problem.

The preferred carbonation reagents are either gaseous carbon dioxide or super critical carbon dioxide. When the carbonation reagent is super critical carbon dioxide, it may be forced into the cohesive product at elevated

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pressures of up to 100 atmospheres. Likewise, when the carbonation reagent is gaseous carbon dioxide, it may be forced into cohesive product at elevated pressures of preferably no less than 30 atmospheres.

For example, the cohesive product from step (c) may be placed in a pressure cylinder and subjected to a vacuum for up to 15 minutes. Thereafter, the cylinder is flooded with water which is then subjected to a positive pressure of typically 6 atmospheres for a further 15 minutes, resulting in water impregnation right through the cohesive product. The cylinder is then drained of water and the cohesive product is subjected to a post vacuum in order to extract from the cohesive product all excess water which "boils" from the cohesive product, as well as to extract any interstitial air. The excess water is drained from the cylinder and compressed carbon dioxide is introduced into the cylinder which penetrates through the cohesive product. The compressed gas feedline may be left open for a period of 30 to 60 minutes in order to allow the various reactions to go to completion. The product is then withdrawn from the cylinder and allowed to dry to provide the finished product in which the Portland cement has substantially set.

Additional hydrophobisation of the finished product may be imposed by including in the water of hydration, a silicone micro emulsion concentrate, dispersed in water at a level of from 0,2 to 8% inclusive by mass, more generally at a level of from 1% to 3% inclusive by mass, thereby forming silicone mesophases. Examples of suitable silicone micro emulsions are Wacker BS1306, Wacker BS1000 and Wacker 1311.

Examples of the invention will now be given.

**Example 1**

Slaked lime	45%
Dicalite 471 (Chemserve perlite)	15%

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RSU.Exfoliated vermiculite (Chemserve vermiculite)	20%
Kaolin 300 mesh	15%
Code 602 novolac resin (Schenectady)	4%
Dolanit PAN fibre	1%
(All percentages by mass).	

The above composition was mixed and then pressed at 180°C to 12 mm thickness and a density of 900 kg/m<sup>3</sup> for 2 minutes, to form a cohesive product. This product was subsequently impregnated with water to a 20% uptake by mass. After a 12 hour humidity drying time, the product was impregnated with super critical carbon dioxide at 70 atm pressure for 8 minutes at 25°C, to give the finished product

#### **Example 2**

Slaked lime	40%
Undensified silica fume	30%
Exfoliated vermiculite with a particle size spectrum of 100 to 300 micron	25%
Novolac resin	5%
(All percentages by mass)	

The above composition was mixed and then pressed at 160°C to a dry density of 1050 kg/m<sup>3</sup> at a thickness of 12 mm to form a cohesive product. This cohesive product was subsequently impregnated in a pressure cylinder with water.

The resulting product was then left for 30 days in a cure chamber and then dried to a water percentage of 2% to give a building board with superior properties.

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## CLAIMS

- 1 A method of making a finished product including the steps of:
    - (a) providing a dry feedstock selected from the group consisting of particles or fibres of a lignocellulosic material; particles of an expanded mineral; particles of a foamed set hydraulic binder; particles of an undensified silica fume; or a mixture of two or more thereof;
    - (b) mixing the feedstock with:
      - (i) a suitable amount of a thermosetting resin in finely divided dry powder form;
      - (ii) a suitable amount of a first binding agent selected from the group consisting of an alkaline earth metal oxide or hydroxide or a mixture of two or more thereof, in finely divided dry powder form;
      - (iii) a suitable amount of a second binding agent selected from the group consisting of:
        - (1) a pozzolan selected from the group consisting of densified silica fume; ground granulated blast furnace slag; fly ash; and a mixture of two or more thereof;
        - (2) an unexpanded clay;
        - (3) a high silicon containing lignocellulosic material;
        - (4) silica;
        - (5) an alkali metal silicate;
        - (6) aluminium oxide or hydroxide;
- to give a starting material;

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- (c) subjecting the starting material of step (b) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form a cohesive product;
  - (d) providing to the cohesive product of step (c) water in an amount sufficient to permit the reaction of the first binding agent with the second binding agent to give a binder; and
  - (e) allowing the binder to set to form the finished product.
- 2 A method according to claim 1 wherein the feedstock is selected from the group consisting of particles or fibres of a lignocellulosic material; exfoliated vermiculite particles; expanded perlite particles; particles of undensified silica fume; and a mixture of two or more thereof.
- 3 A method according to claim 1 or claim 2 wherein the thermosetting resin is a novolac phenol formaldehyde resin.
- 4 A method according to any one of claims 1 to 3 wherein the feedstock is used in an amount of from 10% to 50% inclusive by mass of the starting material, the thermosetting resin is used in an amount of from 1% to 20% inclusive by mass of the starting material, the first binding agent is used in an amount of from 10% to 65% inclusive by mass of the starting material, and the second binding agent is used in an amount of from 10% to 65% inclusive by mass of the starting material.
- 5 A method according to any one of claims 1 to 4 wherein in step (c) the starting material of step (b) is pressed at a temperature of up to 200°C and a pressure of from 20 to 70 kg/m<sup>2</sup> to form the cohesive product.
- 6 A method according to any one of claims 1 to 5 wherein in step (d) simultaneously or sequentially with the provision of water, but before the

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binder has set, there is introduced into the cohesive product of step (c) carbon dioxide to assist in the setting of the binder.

- 7 A method according to claim 6 wherein in step (d) there is added to the cohesive product of step (c) a carbonation reagent selected from the group consisting of carbon dioxide; and an aqueous solution of a compound or compounds which release carbon dioxide; under conditions that cause release of carbon dioxide in the cohesive product.
- 8 A method according to claim 7 wherein the carbonation reagent is selected from the group consisting of super critical carbon dioxide, near super critical carbon dioxide, and high density carbon dioxide which is impregnated into the cohesive product of step (c) under pressure and which provides to the cohesive product of step (c) water in an amount sufficient for the complete hydration of the binder.
- 9 A method according to claim 7 wherein in step (d) there is added to the cohesive product of step (a) water in an amount sufficient for the complete hydration of the binder while simultaneously or sequentially but before the binder has set adding to the cohesive product of step (c) a carbonation reagent which is selected from the group consisting of gaseous carbon dioxide, liquid carbon dioxide, super critical carbon dioxide, or a solution of carbon dioxide in a solvent.
- 10 A method according to claim 7 wherein in step (d) there is added to the cohesive product of step (c) a carbonation reagent selected from the group consisting of a compound which releases carbon dioxide; and compounds which together release carbon dioxide; as a solution in water in an amount sufficient for the complete hydration of the binder.

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- 11 A method according to claim 10 wherein the carbonation reagent is selected from the group consisting of potassium carbonate and sodium carbonate.
- 12 A method according to claim 6 wherein in step (b) there is added to the starting material a carbonation reagent which is a dry compound or dry compounds which release carbon dioxide under specified conditions, and in step (d) subjecting the cohesive product of step (c) to the specified conditions to cause the release of carbon dioxide in the cohesive product.
- 13 A method according to claim 12 wherein the carbonation reagent is selected from the group consisting of potassium carbonate and sodium carbonate.
- 14 A finished product made by the method of any one of claims 1 to 13.